JAPANESE [JP,2003-268354,A]

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the manufacturing method of the fumed silica aquosity dispersion liquid which can be used as polishing slurry etc. which are used by the manufacturing process of a semiconductor or electronic parts.
[0002]
[Description of the Prior Art]The fumed silica particles conventionally compounded by using a silicon tetrachloride, hydrogen, and oxygen as a raw material by the vapor phase hydrolysis reaction (fumed **) in the inside of a high temperature flame are known.
[0003]Said fumed silica is a high grade raw material with very few impurities, and the use is various, for example, is used also as an abrasive grain of semiconductor industry-oriented abrasive soap slack aquosity dispersion liquid (polishing slurry of a semiconductor wafer). The aquosity dispersion liquid of this fumed silica distribute silica dry type powder first in pure water (or pure water including a chemical), and are formed by adding basic liquid in these silica dispersion liquid, and adjusting pH to basicity.
[0004]However, where these aquosity dispersion liquid are settled, fume silica tended to maintain a good dispersion state, but when it continued giving continuous energies, such as shake and circulation, there was a problem that a dispersion state will become unstable and will condense finally.
[0005]
[Problem(s) to be Solved by the Invention]Then, even if this invention continues giving energy continuously, it tends to provide the manufacturing method of the fumed silica

Drawing selection Drawing 1

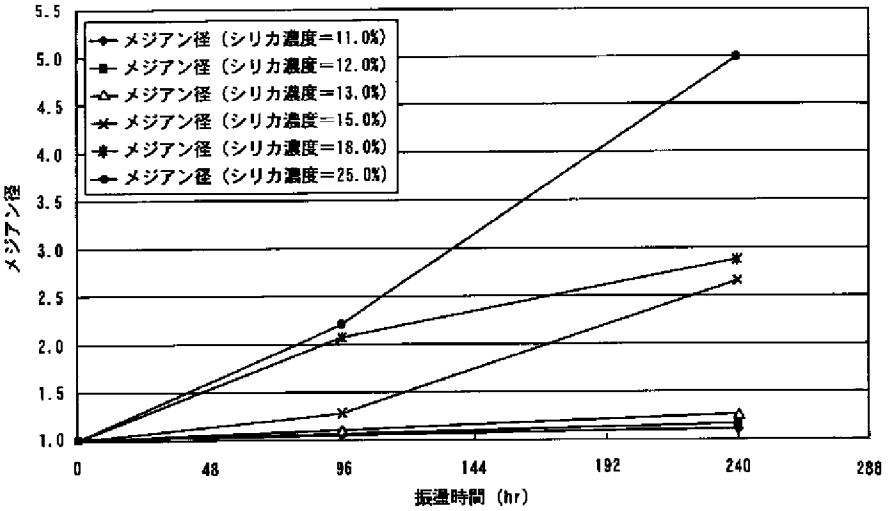

Fig.Shaking test results（メジアン径）

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aquosity dispersion liquid which a silica particle does not condense easily conventionally.
[0006]
[Means for Solving the Problem]In order to solve said technical problem, the following technical means are provided in this invention.
[0007]** A manufacturing method of fumed silica aquosity dispersion liquid of this invention, By adding fumed silica, forming middle aquosity dispersion liquid in which said silica concentration is about 20 to 60 % of the weight, while pH applies shearing force into liquid of about 2-11, and adding said middle aquosity dispersion liquid into basic liquid, It adjusted so that obtained silica concentration might be about 1 to 50 % of the weight and pH might be set to about 8-12.
[0008]In this manufacturing method, since middle aquosity dispersion liquid of fumed silica were added into basic liquid, Since fumed silica is diffused every in small quantities into basic liquid in a process in which silica dispersion liquid are added and silica concentration in basic liquid (it becomes an "acceptance parent") increases gradually from zero, F-potential can shift to an alkaline region which becomes it is high and stable [a dispersion state] quickly, and the silica particle in middle aquosity dispersion liquid to add can improve stability to condensation of dispersion liquid. According to this method, in the case of addition of middle aquosity dispersion liquid, condensation does not take place easily by a grade which performs loose stirring.
[0009]By a method of adding into middle aquosity dispersion liquid of fumed silica, like before basic liquid. A silica particle of middle aquosity dispersion liquid (it becomes an "acceptance parent") of fumed silica will shift to an alkaline region through a neutral region from acidic regions where F-potential is low and a dispersion state is unstable, and if stirring which gives sufficient shearing force in the case of addition of basic liquid is not carried out, it will be very easy to condense it.
[0010]** It may be made for obtained silica concentration to be about ten to 30 wt%.
[0011]About ten to 30 wt%, silica concentration obtained in this way can raise stability to condensation more, preferably if about ten to 20 wt%.
[0012]** It may be made for obtained silica concentration to be about 11 to 13 wt%.
[0013]If constituted in this way, a growth rate of median particle diameter is about 50% or less by shaking examination of 240 hours of dispersion liquid. With said shaking examination, 20 ml of slurries are put into a 50-ml centrifugation tube here, They are the contents of setting to a vertical mold shaker, and shaking speed beginning by 300spm, and a shaking stroke starting an examination at 40 mm, removing a centrifugation tube after 240-hour progress, measuring median particle diameter of dispersion liquid, and comparing shake before.
[0014]** After adding said middle aquosity dispersion liquid into basic liquid, it may be made to pass a filter with an aperture of about 30 micrometers or less.
[0015]When constituted in this way, there is an advantage that what is called a coarse particle which is particles and floc to which distribution in a slurry was not fully performed

is removable.
[0016]** It may be made for fumed silica to add to have the specific surface area of about 40-300 square m/g.
[0017]When constituted in this way, it becomes possible to control a size of a silica particle in silica dispersion liquid obtained as mentioned above, and there is an advantage that it is possible to adjust performance (for example, polishing speed) as abrasive soap.
[0018]** As a substance which adjusts pH, chloride, nitric acid, sulfuric acid, phosphoric acid, etc. can be used for adjustment by the side of acidity, and ammonium hydroxide, sodium hydroxide, a potassium hydrate, calcium hydroxide, barium hydroxide, magnesium hydroxide, etc. can be used for adjustment by the side of alkali.
[0019]** As an alkali which adjusts the pH of fumed silica aquosity dispersion liquid, ammonium hydroxide, sodium hydroxide, a potassium hydrate, calcium hydroxide, barium hydroxide, magnesium hydroxide, etc. can be used.
[0020]
[Embodiment of the Invention]Hereafter, this embodiment of the invention is described.
[0021]While pH applies shearing force into the liquid of about 2-11 (for example, ultrapure water), the manufacturing method of the fumed silica aquosity dispersion liquid of this embodiment adds fumed silica, and he is trying to first form the middle aquosity dispersion liquid in which said silica concentration is about 20 to 60 % of the weight.
[0022]and the silica concentration obtained by adding said middle aquosity dispersion liquid into basic liquid (for example, ammonia liquor) -- about 1 to 50 % of the weight -- and it adjusts so that pH may be set to about 8-12.
[0023]In this manufacturing method, since the middle aquosity dispersion liquid of fumed silica were added into basic liquid, Since fumed silica is diffused every in small quantities into basic liquid in the process in which silica dispersion liquid are added and the silica concentration in basic liquid (it becomes an "acceptance parent") increases gradually from zero, F-potential can shift to the alkaline region which becomes it is high and stable [a dispersion state] quickly, and the silica particle in the middle aquosity dispersion liquid to add can improve the stability to condensation of dispersion liquid.
[0024]Therefore, even if it continues giving energy continuously, there is an advantage of being hard to condense a silica particle conventionally.
[0025]Next, the composition of this invention is explained more concretely.
(Preparation of fumed silica aquosity dispersion liquid) 21 kg of ultrapure water is thrown in in a mixer tank (50L capacity), 9 kg of fumed silica (70 square m/g of specific surface areas) is added one by one, applying shearing force to said ultrapure water, and the middle aquosity dispersion liquid whose fumed silica concentration is 30wt% were generated.
[0026]And said generated middle aquosity dispersion liquid are gradually added in ammonia liquor, and final fumed silica concentration prepared the fumed silica aquosity dispersion liquid in which pH is set to 10-11 at 25wt% 18wt % 15wt% 13wt% 12wt% 11wt%.
(Shaking examination) The next shaking examination

estimated condensation stability using the fumed silica aquosity dispersion liquid of each of said concentration. That is, 20 ml of samples of each fumed silica aquosity dispersion liquid were put into a 50-ml centrifugation tube, it set to the vertical mold shaker (Iwaki industrial company make, form name KMShaker V-DX), and shaking speed started shake by 300spm and 40 mm of shaking strokes.

[0027]After starting shake, after the predetermined passage of time, the centrifugation tube was removed (to 240-hour progress), the median particle diameter of each sample was measured, and the path before shake was compared as the standard 1. The graph of the relation between the growth rate of the median size of fumed silica aquosity dispersion liquid and shaking time is shown in drawing 1.

(Result) The growth rate of median particle diameter was so stable few that fumed silica concentration was low as shown in the graph of drawing 1. Especially, also in the shaking examination of 240 hours, the growth rate of median particle diameter is less than 50%, and, as for the sample not more than 13wt%, fumed silica concentration showed very remarkable stability.

[0028]These fumed silica aquosity dispersion liquid can be used, for example as semiconductor industry-oriented abrasive soap slack aquosity dispersion liquid (polishing slurry of a semiconductor wafer) etc.

[0029]

[Effect of the Invention]This invention is the above composition and has the following effect.

[0030]Since the stability to condensation of dispersion liquid can be improved, even if it continues giving energy continuously, the manufacturing method of the fumed silica aquosity dispersion liquid which a silica particle does not condense easily conventionally can be provided.

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